Application of Cold Plasma to Improve Properties of Phenolic-Bonded Aspen Fiberboard

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ABSTRACT. Aspen fibers were plasma activated to enhance bonding between the particles and the adhesive for wood composites. The activation of materials was carried out by environmental-friendly dry plasma processing. After argon, oxygen or ammonia plasma treatments, the aspen fibers were analyzed by ESCA and FTIR. The fibers were used for designing fiberboards. Mechanical (MOR and MOE) and water swelling tests were conducted to evaluate the properties of aspen fiberboards. The results showed that ammonia and oxygen-plasma treatments of virgin and extracted aspen fiber samples have been the most successfully for surface modification and improving the strength of the fiberboards.

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KEYWORDS. Fiberboard, cold plasma, surface modification, wood particles, lignin

INTRODUCTION

Medium density fiberboard is usually manufactured without any chemical or physical treatments to the fiber before the board is formed. A small percentage of wax can be added to improve water repellency. It is possible to modify the fiber prior to board formation to greatly improve performance of the finished product. Heat treatments, chemical modification, and enzyme treatments have been used for this purpose with very positive results. These methods, in general, change the chemistry of fiber through out the board and, therefore, change bulk properties of the fiberboard (Winandy and Rowell, 2005; Rowell, 2005).

Cold plasma technology can be used to change just the surface characteristics of the fiber before board formation. Interfiber and intrafiber bonding can be increased by functionalizing the surfaces of the fibers, which may result in increased water repellency and wet strength.

Covalent bonding of desired functionalities to specific natural and synthetic substrate surfaces is a key element for the development of "within-interphase" molecular interaction, which is required for the preparation of advanced composites. Cold-plasma chemistry offers novel solutions, not only in the area of functionalization of even the most inert organic and inorganic polymeric substrates but it also opens up new routes for heterogeneous (solid/gas)-dry-chemistry mediated reaction mechanisms, by taking advantage of both the neutral and charged active species coexistent in the discharge (Timmons & Griggs, 2004; Denes & Manolache, 2004; Denes et al., 2005).

The electron energy distribution of non-equilibrium (low-pressure) plasmas can be often described by a Druyvesteyn approximation, which indicates that a small number of electrons have relatively high energies (5–15 eV) while the bulk of the electrons belong to the low-energy electron range (0.5–5 eV). Since the ionization potentials of the atoms of common organic structures belong to the tail region of the electron energy distribution e.g., $C^+ = 11.26$ eV; $H^+ = 13.6$ eV; $O^+ = 13.6$ eV; $N^+ = 14.53$ eV, etc., the low degrees of ionization of cold plasmas appear obvious. However, it is important to note that the energy range of most of the electrons (2–5 eV) is intense enough to dissociate almost all chemical bonds involved in organic structures and to create free radical species capable of reorganizing into a variety of structures.

As a consequence, all organic derivatives and organic compounds containing main group elements can be converted into high molecular weight compounds, even if they do not have the peculiar functionalities for common monomers.

Radio frequency (RF) reactors are most commonly used for synthesis and surface modification of various substrates. These discharges can be excited and sustained even by using insulated electrodes located inside or outside the reaction chamber. RF plasmas can also be sustained under lower pressure conditions and have higher ionization efficiencies than DC discharges. The energies of sample-bombarding ions of RF plasmas can conveniently be controlled by adjustable self-bias while, in the case of DC plasmas, this possibility is limited by the breakdown-voltage. The advantages of RF plasma chemistry approaches can be summarized as follows.

- It involves only a very thin surface layer of the substrates exposed to the discharge (around 10 nm into plasma-enhanced modification processes; the bulk of the material remain practically unmodified).
- It is dry chemistry and accordingly it do not require the use of large amounts and often toxic of organic chemicals that carry potential environmental hazards.
- The energy levels of plasma species are comparable with the common bond energies, and as a consequence even the most inert material surfaces can be conveniently altered.

Extractives on the surface of the fiber may influence fiber bonding so experiments have been done using both non-extracted and solvent extracted fibers. Aspen has a very low content of extractives (less than 2%); however, a part of its extractive components is located on the fiber surfaces, and due to the fact that plasma-enhanced modifications are *par essence* surface reaction mechanisms, the effect of these extractives on inter- and intra-fiber bonded is evaluated. The ultimate goal of cold plasma modification of fibers is to use this technology to bond fibers together without the use of any added adhesive.


EXPERIMENTAL

Materials

Green aspen logs from Northern Wisconsin were hand debarked, chipped, and refined at USDA Forest Products Laboratory-Madison pilot plant.

Refining was performed using a Sprout-Bauer 12–1 CP 12 inch (30.5 cm) pressurized refiner. Chips were presteamed at 90 psi (166°C) for 10 min and then refined under pressure. After refining, the fibers were air dried and extracted with Toluene: Ethanol solvent (1:2.33 ml ratio). Furfuryl alcohol, phenol-formaldehyde, as well as toluene and ethanol were purchased from Aldrich Chemical (Milwaukee, WI). Conventional adhesives were obtained from standard suppliers. Argon, oxygen and ammonia used as gas media for plasma-enhanced reactor-cleaning operations and plasma treatments of aspen fibers were obtained from Liquid Carbonic (Brookfield, WI).

Argon, oxygen and ammonia were purchased from AGA-Linde (Wilmington, DE). Fluorescamine was obtained from Molecular Probes Inc., (Eugene, OR), and ethanol and toluene from Aldrich Co.

Methods

The presence of primary amine functionalities on functionalized PE and glass surfaces was evidenced by fluorescence labeling technique. Fluorescamine reacts with primary amine groups and generates a complex according to the reaction scheme:
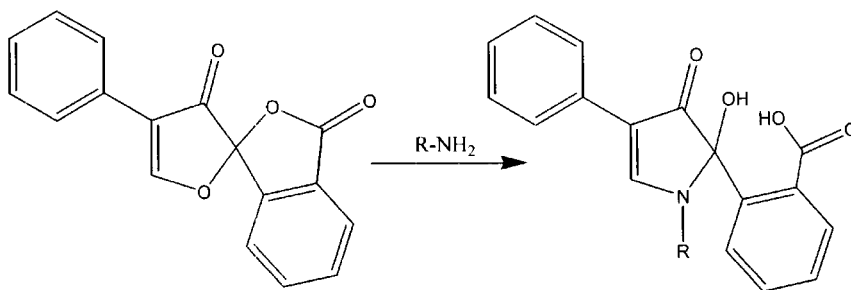
The complex absorbs at 381 nm UV radiation and emits at 470 nm in the blue region.

Ammonia-plasma-treated aspen samples were sprayed 3 times consecutively with a fluorescamine solution (25 mg fluorescamine in 100 ml acetone) by using a Gelman Chromist aerosol propellant, attached to polypropylene bottle. The fluorescence of the substrates was revealed with the aid of a Black-Ray UV-lamp, model: UBL 21 (UVP Inc. San Gabriel, CA) and a FCR-10 photo camera (Fotodyne Inc., Hartland, WI).

Evaluation of Binding Energy (BE) Shifts for ESCA Data

All virgin and modified aspen samples were 3 seconds gold-coated using a Desk II instrument from Denton Vacuum Inc. The BE shift corrections were performed using the 83.9 eV Au-4f$_{7/2}$ BE value.

Extraction of Aspen Fibers

Wood extractives are materials soluble in neutral solvents and are not generally considered as part of the wood substance. These materials should be removed before any chemical analysis of wood substance, except where the extraction process and subsequent washing could interfere with certain chemical analyses.

Ethanol-toluene was used to extract waxes, fats, some resins, and probably some portions of wood gums. The solvent used for extraction was a mixture of ethanol-toluene (2.33:1 ml volumetric ratio). The ethanol-toluene extraction lasted 5 h. After extraction, alcohol was used to remove catechol tannis and toluene from the aspen fibers.

Board Manufacture and Testing

The untreated and plasma treated fibers were sprayed with the appropriate adhesive formulation (44% solution of conventional phenol-formaldehyde resin, such as Plenco 650 phenol resin) in a rotary blender. The material was then formed into a 6-in square forming box and pressed at an average 400 psi for 30 min between hot plates (400°F) to produce 0.125 inch thick boards with a density of about 0.60 g/cm^3. The press used in all preparations was a Carver press from USDA Forest Products Lab, Madison.

To evaluate the modulus of rupture (MOR) and modulus of elasticity (MOE), the 6×6 inch boards were cut into 6×2 in. For mechanical testing of the boards an Instron 5544 instrument was employed at the USDA Forest Products Laboratory-Madison.

For performing the water soak tests the boards were cut into 2*2 inch and oven- dried for 24 h. After drying, the thickness of the boards was measured in 4 arbitrary points. The dried boards were placed into a dish with water for 24 h, and then the thickness was measured again at the same four geometrical locations. Testing results were compared to control samples run in the absence of plasma treatment.

Analytical Techniques (ESCA and ATR-FTIR)

The relative surface atomic concentrations and the C1s, O1s and N1s high resolution peaks corresponding to atoms in non-equivalent positions of aspen and modified aspen samples were analyzed using a Perkin Elmer Physical Electronics 0 5400 small area ESCA system (Mg source; 15 kV; 300 W; pass energy: 89.45 eV; angle). In order to correct surface-charge-origin binding energy shifts calibrations were performed based on gold ($4f_{5/2}$: 87.6 eV and $4f_{7/2}$: 83.9 eV) sputtered on sample surfaces.

Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR-FTIR) spectroscopy measurements from untreated and plasma-modified natural polymeric substrates were carried out using a ATI-Mattson, Research Series instrument, provided with an ATR unit. High resolution ATR-FTIR evaluations were performed under nitrogen blanket generated fro a flow-controlled liquid nitrogen tank. Data were collected in the 600–4000 cm^{-1} wavenumber region with **250** scans for each sample.

Plasma Modification of Wood Fibers

An original 13.56 MHZ-RF-plasma reactor was designed and developed (Figure 1) for surface modification of natural and synthetic polymeric substrates with particulate and fiber morphologies. The reactor is composed of a Pyrex glass chamber (7) provided with connecting rubber and stainless steel rings (5, 9) on both ends. The vacuum-tight connection of the reactor to the monomer- and gas-supply system and to the vacuum line is assured with the aid of two ferrofluidic feed-throughs (4, 10). The hollow shaft of the stainless steel chambers (4, 10) are made of special magnetic material and they are a part of the ferrofluidic sealing system. The RF power is transferred to the reactor through two semi-cylindrical cooper electrodes (6) located outside from a 1000 W, 13.56 MHZ RF power supply and matching network assembly (8). A large cross-section gate valve (11) separates the reactor from the vacuum line and allows the control of the out-flow of plasma-gases. The vapor and gas flow into the reactor is controlled through individual flow controllers (3). The rotation of the reactor at various angular velocities is assured by a digitally speed-controlled electric engine-transmission system (12, 13).

The following experimental conditions are used for the plasma treatments in the rotary reactor: substrates, wood fibers; grafting monomer, ethylenediamine; plasma gases, Ar, O_2, NH_3; RF power dissipated to the

FIGURE 1. Schematic diagram of rotating RF-plasma reactor.
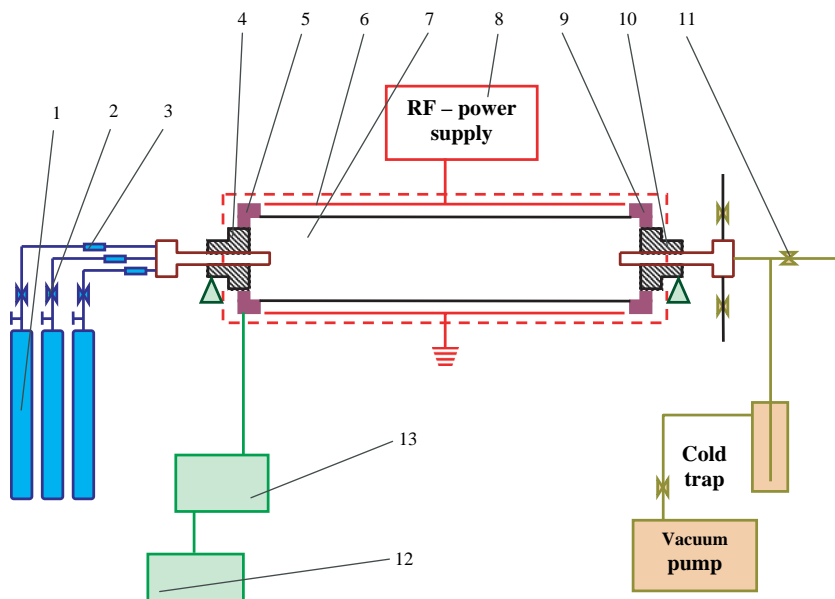
electrodes, 100–250 W; base pressure, 40–50 mTorr; pressure in the absence of plasma, 390 mTorr; gas flow rate, 20–22 sccm; treatment time, 10, 15 min; angular speed, 30 rpm.

RESULTS AND DISCUSSION

The relative surface atomic concentrations of aspen samples are presented in Table 1. It can be observed that the high carbon concentration associated with the non-extracted and non-plasma treated fibers is significantly reduced and the oxygen concentration is increased as a result of the extraction process. It is known that extractives, composed mainly of steryl esters, waxes, and so on, of high carbon content, usually result in pitch formation during pulping, migrate during wood processing, and storing on the surface. The removal of these compounds by extracting the aspen samples is considered responsible for the dramatic relative surface atomic composition changes. It was also found that ammonia-plasma exposure of non-extracted aspen substrates results in significantly lower relative surface

TABLE 1. Relative surface atomic concentrations of untreated
and NH$_3$-plasma treated aspen fibers (none coated)

Aspen fibers	Carbon (%)	Oxygen (%)	Nitrogen (%)	C/N	C/O ratio
Aspen (none extracted & none treated)	82.8	17.2	—	—	4.8
Aspen (extracted & none treated)	64.5	35.5	—	—	1.8
NH$_3$-plasma aspen (none extracted)	75.7	12.8	11.5	6.5	5.9
NH$_3$-plasma aspen (extracted)	61.9	26.3	11.9	5.2	2.3
O$_2$-plasma aspen (none extracted)	67.3	32.7	—	—	2.0
O$_2$-plasma aspen (extracted)	56.9	43.1	—	—	1.3

nitrogen atomic concentration in comparison to the extracted and plasma treated samples. It is suggested that simultaneous surface functionalization and etching the lower molecular weight compounds located on the sample surfaces (e.g., extractives) render lower nitrogen atomic concentrations.

Early attempts to anchor primary amine functionalities onto polymer surfaces using cold-plasma approaches involved ammonia-gas environments. Later on, due to the low efficiencies of ammonia-origin, primary amine group implantation reactions, and saturated and non-saturated linear and aromatic amines were employed as primary amine precursors.

Comparing the bond energies and enthalpies of formation of free radicals of some of the N$_x$H$_y$ species one can observe that the N-H bond energy of ammonia (4.66 eV) is significantly higher in comparison to other poly-atomic species and that of the diatomic species (3.51 eV), and that the enthalpies of formation of NH (3.65 eV) is much higher in comparison to those of NH$_2$ and N$_2$H$_3$ (1.91 and 2.52 eV) radicals. This indicates that higher average electron energy is required for initiation of the dissociation of NH$_3$; however, this energy level is too intense for the "survival" of NH$_2$ species. The majority of the cold plasma environments are characterized by average electron energies of 2–4 eV; consequently, most of the species generated under NH$_3$-plasma environments should result in a predominant NH free radical formation. Covalently anchored >NH functionalities onto various polymer surfaces, undergo in open laboratory conditions hydrolysis that results in the formation of surface C=O groups and ammonia. Accordingly, it is not surprising that the nitrogen content of the ammonia-plasma treated aspen fibers is diminished during the storing process. However, it is assumed that the simultaneously generated =C=O groups on the aspen fiber surfaces will play a significant role in an improved interphase adhesion mechanism of the fibers.

High resolution C1s and O1s ESCA data substantiate the conclusion that the ammonia-plasma-modified fiber surface-layers are composed mainly of various carbon- and oxygen-based bonds, and that the surface concentration (areas of BE peaks) of C/O functionalities increased as a result of modifications in the presence an absence of plasma. It is noteworthy that the nitrogen content of the ammonia-plasma-treated samples diminishes in time under OLC that indicates the presence of NH-groups on the discharge-modified surfaces. It is well known that NH (imine) functionalities hydrolyses in the presence of moisture with the generation of $C=O$ groups and ammonia. Table 1, exhibits the relative surface areas of BE peaks corresponding to C and oxygen atoms incorporated in non-equivalent atomic environments.

Fibers that were analyzed immediately after ammonia-plasma treatment indicated that the extracted and non-extracted fibers have comparable surface amino-group concentrations.

Table 1, shows that after solvent extraction, there is a significant change in the carbon and oxygen content of the wood surface. Extractives in wood tend to migrate to the surface. Since plasma treatments are confined to the surface, it was of interest to study the natural surface as well as a surface with the extractives removed. Table 2 shows that removing the surface extractives has little beneficial effects on swelling properties of the fiberboard.

Figure 2, a and b exhibit the C1s and O1s high resolution (HR) spectra of non-extracted and non-plasma-treated aspen fibers. The presence of dominant surface area C-C and $C=C$ BE peaks is evident in addition to a much smaller surface area C-O peak, that is indicative for the presence of extractive molecules on the surface in addition to lignin. The O1s diagram shows besides the presence of C-O (533.5 eV) and C-O-C (532.9 eV) functionalities characteristic to lignin and cellulose also the existence of aromatic ketone and ester type functionalities (531.2 eV). ESCA measurements performed at 45° take

TABLE 2. Water swelling results for aspen fiberboards

Aspen fibers	Average water swelling (%)
Aspen (none extracted & none treated)	26
Aspen (extracted & none treated)	40
NH_3-plasma aspen (none extracted)	26
NH_3-plasma aspen (extracted)	40
O_2-plasma aspen (none extracted)	19
O_2-plasma aspen (extracted)	31

FIGURE 2. High resolution C1s and O1s ESCA diagrams of non-extracted and non-treated aspen fibers.

a.

b.
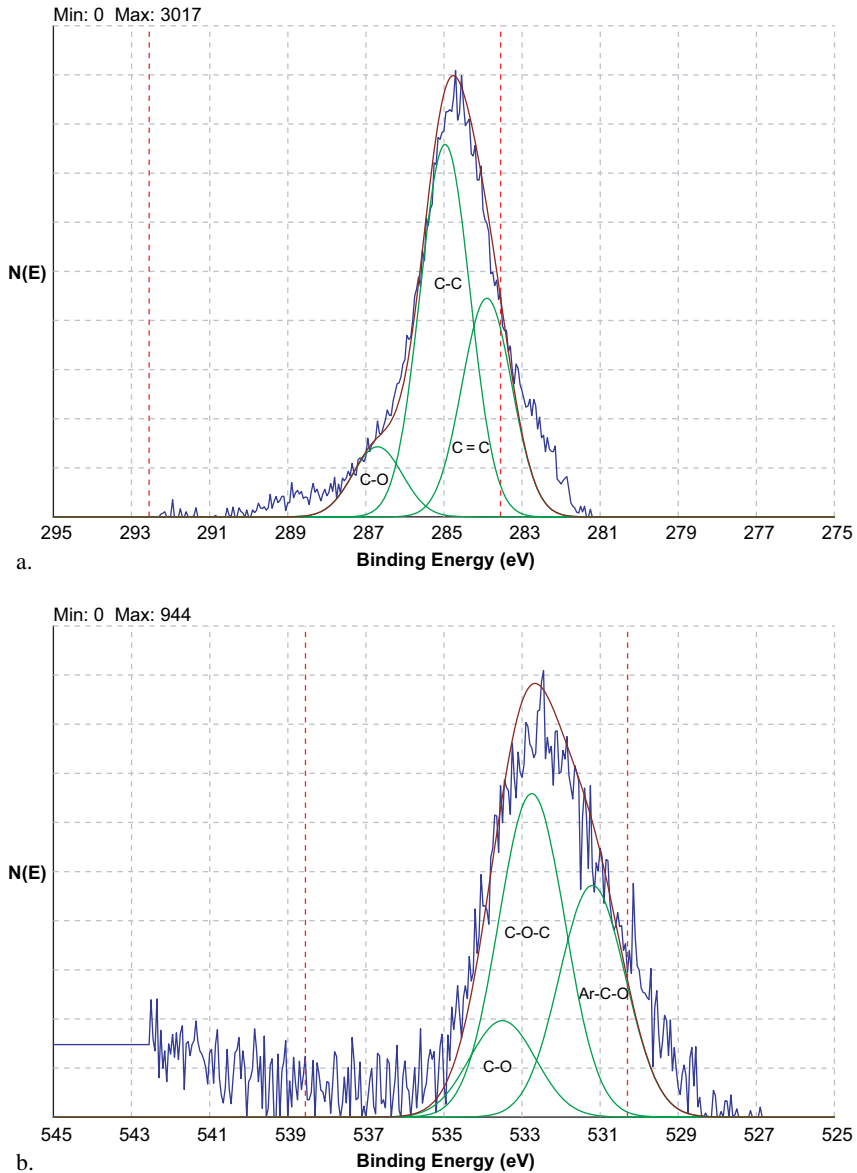
of angle give information on the top 10-nm surface layer and as a consequence, due to the thin layer nature of the extractive layer lignin and cellulose substrate-origin information is also incorporated into the spectra.

As a result of the extraction, both the surface areas of C-C and C=C peaks versus the C-O and O-C-O peak areas and the surface areas of ketone- and ester-type BE peak areas versus the C-O peak area decreased notably (Figure 3a,b and Table 3). These results are also indicative for the removal of extractives and for the surface-exposure of lignin and cellulose structures.

HR ESCA diagrams of non-extracted and ammonia-plasma-treated aspen fibers (Figure 4a,b) show little differences relative to the diagrams of non-extracted and non-plasma-treated samples. However, the presence of a decreased C=C BE peak area relative to the C-C/C-N peak area allows us to suggest that a plasma-induced surface etching reaction mechanism took place with a simultaneous, additional incorporation of C=O (288 eV) functionalities (Table 3).

C1s ESCA diagram of extracted and ammonia-plasma-treated aspen fibers (Figure 5a) is similar to the ESCA spectrum of extracted aspen samples (Figure 4a). Due to the very close binding energy values of C-C (285 eV) and C-N bonds (285.5 eV) deconvolution of the corresponding broad peak area would only be speculative. However, the O1s diagram of plasma modified fibers (Figure 5b) indicates the presence of a slightly increased peak area that is characteristic to aromatic ketone and ester functionalities relative to the C-O peak area. NH-groups-mediated oxidation reactions are suggested to be responsible for these differences (Table 2).

ATR-FTIR data collected from extracted and extracted and ammonia-plasma-treated sisal substrates substantiate the ESCA results that mainly NH functionalities were implanted onto the substrate surfaces as a result of the ammonia plasma exposure. Figure 6a shows the high resolution ATR-FTIR diagram in the 3000–3700 cm^{-1} wavenumber region of the extracted aspen sample. The presence of a broad absorption in the 3200–3520 cm^{-1} domain allow us to suggest that both inter- (3200–3400 cm^{-1}) and intra- (3450–3520 cm^{-1})-molecular OH-based hydrogen bonds are present in the surface layers of the samples. The absence of any significant absorption in the 1500–1700 cm^{-1} wavenumber zone should be noted (Figure 6b).

The existence also in the IR spectrum of the ammonia-plasma-treated sisal substrates (Figure 7a) of a broad absorption band in the same region (3200–3600 cm^{-1} is not surprising because NH stretching modes of primary and secondary amides and stretching and deformation modes of primary and secondary amines are located in the same wavenumber

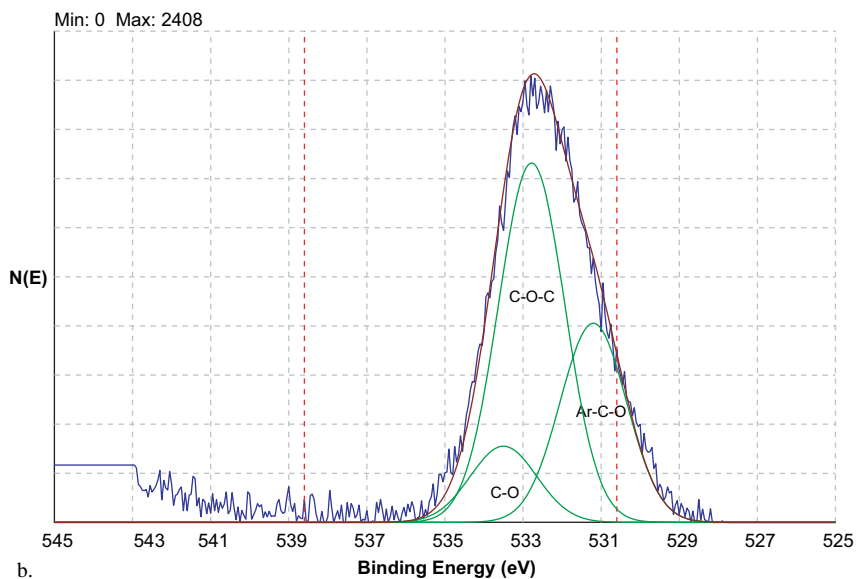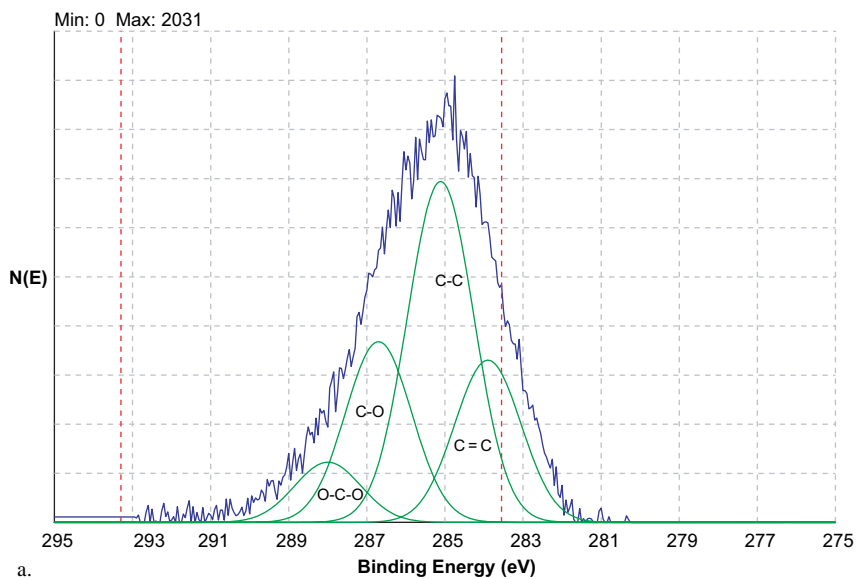FIGURE 3. High resolution C1s and O1s ESCA diagrams of extracted and non-treated aspen fibers.

a.

b.

TABLE 3. C1s and O1s deconvoluted binding energies peak areas

Sample	Carbon peak (%)				Oxygen peak (%)		
	285.0	283.9	286.7	288.0	532.9	531.2	533.5
Non-extracted & none treated aspen	56.3	33.1	10.6	–	48.5	36.4	15.1
Extracted & none treated aspen	47.6	19.5	23.1	9.8	56.9	31.2	11.9
NH$_3$-plasma aspen (none extracted)	56.3	22.6	15.3	5.8	52.6	33.2	14.2
NH$_3$-plasma aspen (extracted)	46.9	18.2	26.3	8.5	53.1	37.5	9.3
O$_2$-plasma aspen (none extracted)	48.4	26.7	16.2	8.7	56.1	28.7	15.2
O$_2$-plasma aspen (extracted)	45.3	23.9	24	6.9	55.5	35.8	8.8

regions with the OH group vibrations, as well. However, a sharp differ-ence can be noted between the 1500–1750 cm^{-1} wavenumber domains of the extracted and extracted and ammonia-plasma modified aspen samples. The presence of a strong absorption in the 1550–1680 cm^{-1} wavenumber region of the IR spectrum of the ammonia-plasma-modified aspen substrates (Figure 7b), that is characteristic of NH deformation vibrations of primary and secondary amines (1550–1650 cm^{-1}) and CO absorptions of primary and secondary amides (1650–1680 cm^{-1}), and the absence of any significant vibration in the same wavenumber region of the extracted aspen samples (Figure 7b) is indicative for the ammonia-plasma-enhanced implantation onto the aspen substrate surfaces of amine and amide functionalities.

It is noteworthy that the fluorescamine test performed on extracted and ammonia-plasma-treated samples compared to the extracted and non-plasma-treated samples a significantly enhanced brightness. This is indicative for the absence of primary amine functionalities or the presence of very low surface primary amine group densities (Figure 8).

Survey N1s ESCA data resulting from extracted and Ammonia-plasma treated substrates that were analyzed consecutively to the plasma treatment and samples that were stored for 3 weeks under OLCs, only shows a slight decrease in the relative surface nitrogen atomic contents (N content consecutive to the plasma treatment: 11.9%; nitrogen content after storing the plasma treated samples for 3 weeks: 7.2%). This is good agreement with the fluorescamine results, which indicate the presence of very low-surface primary amine concentrations.

Relative surface atomic composition data resulting from ammonia-plasma-treated samples stored under OLCs show a slightly diminished nitrogen atomic concentration both for the extracted and non-extracted substrates. It is suggested

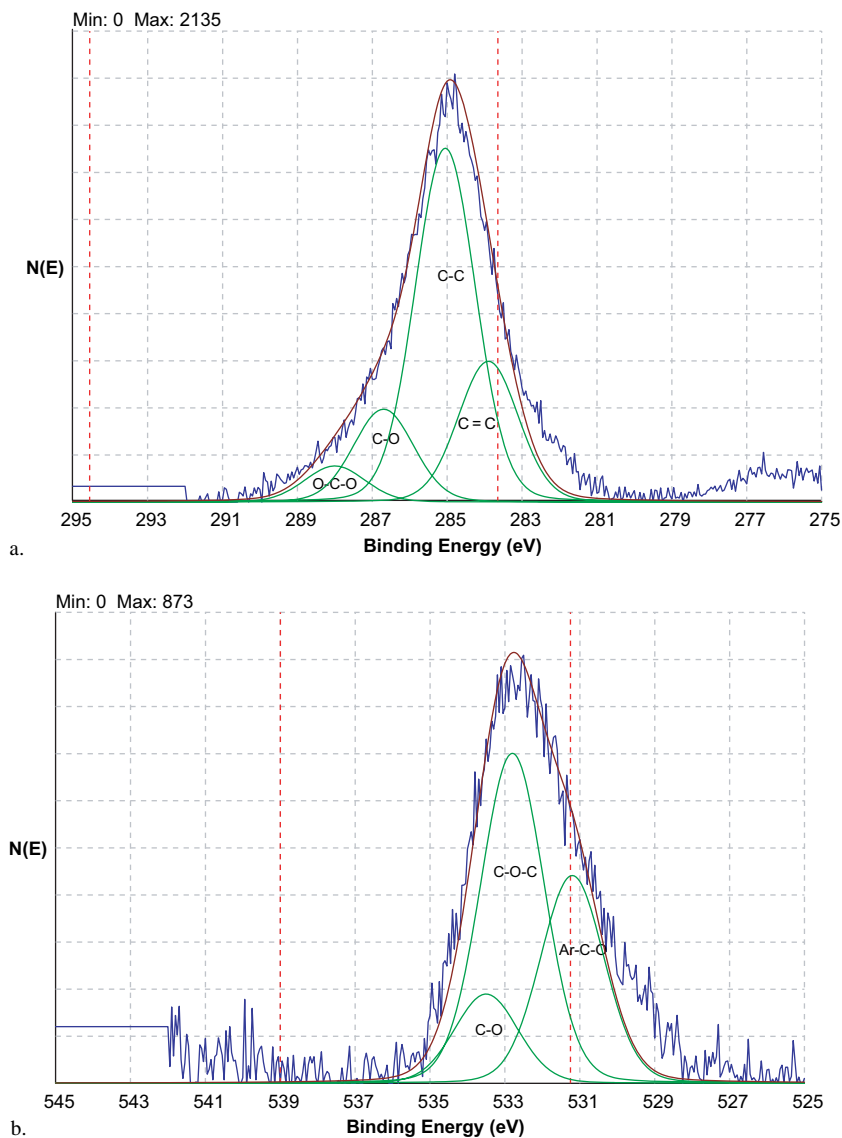FIGURE 4. High resolution C1s and O1s ESCA diagrams of NH$_3$-plasma treated aspen fibers (non-extracted).

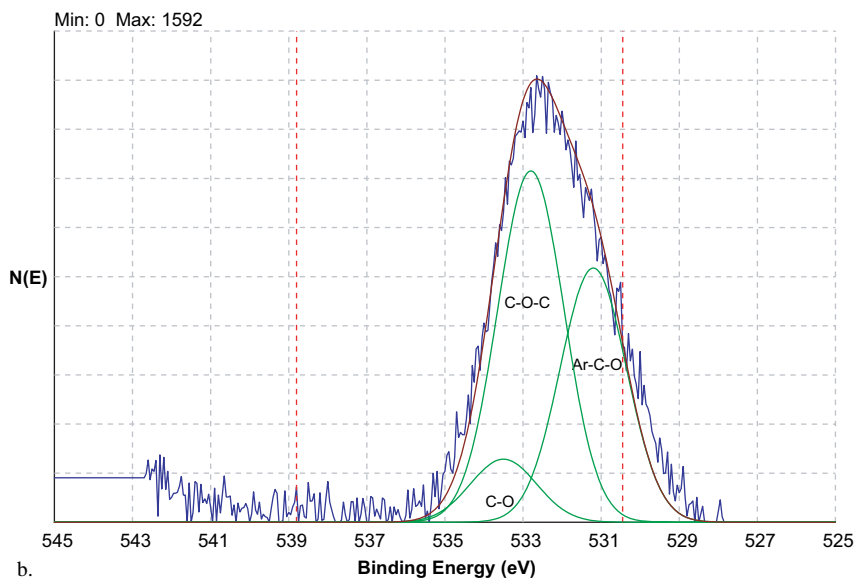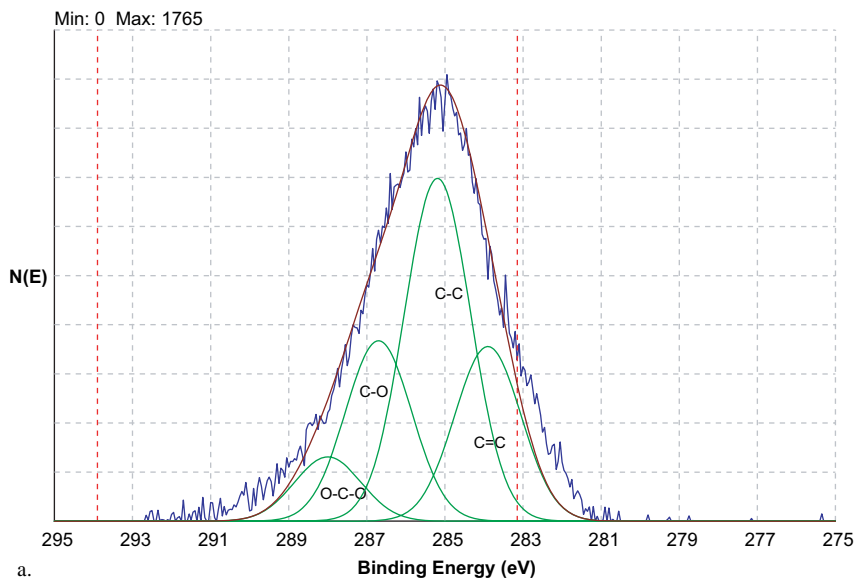FIGURE 5. High resolution C1s and O1s ESCA diagrams of NH_3-plasma treated aspen fibers (extracted).

a.

b.

FIGURE 6. FTIR spectrum of extracted aspen particles. Wavenumber ranges: (a) 3700–3000; (b) 1800–1500.
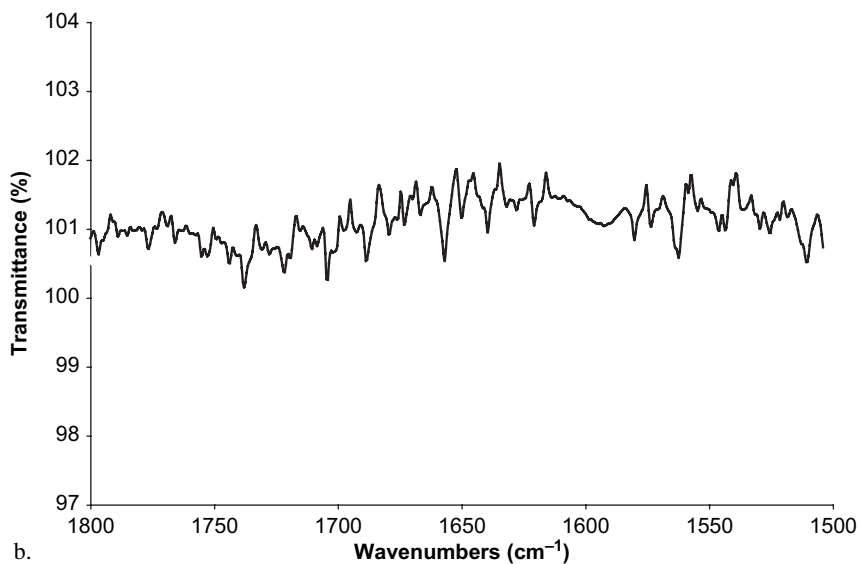
a.

b.
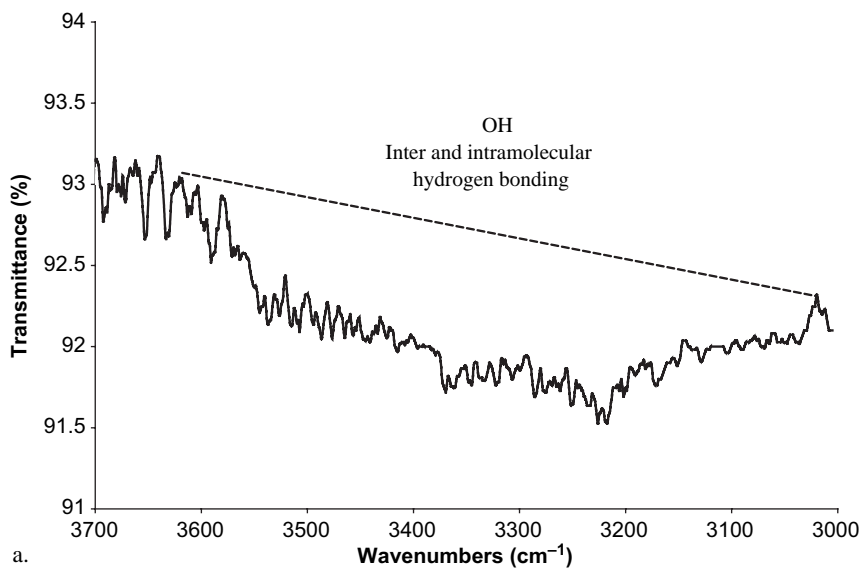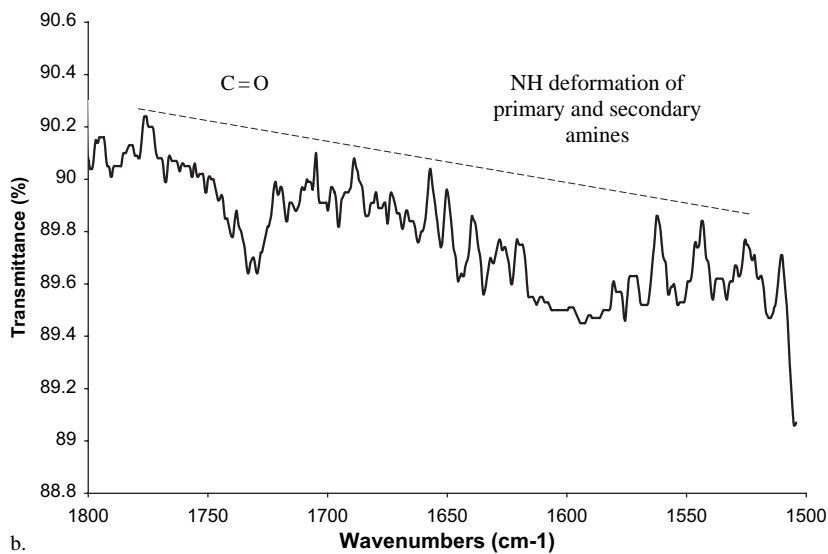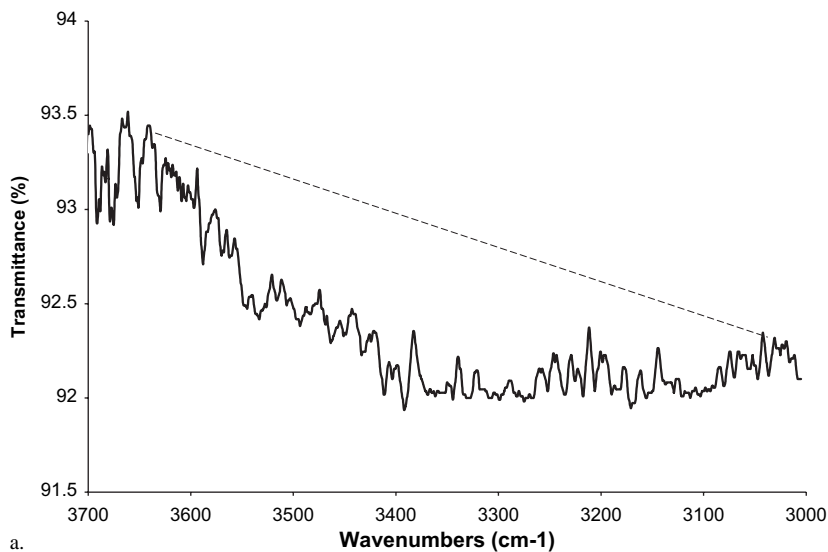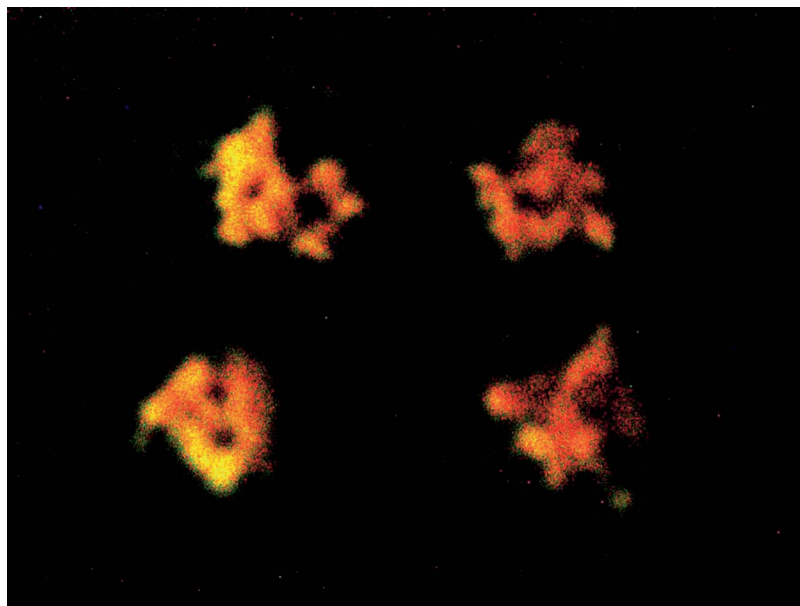
FIGURE 7. FTIR spectrum of NH$_3$-plasma treated aspen fibers (extracted).

FIGURE 8. FTIR spectrum of O_2-plasma treated aspen fibers (extracted).

Upper left: extracted and non-treated;
Upper right: non-extracted and non-treated;
Lower left: extracted and NH_3-plasma treated;
Lower right: non-extracted and NH_3-plasma treated.

that part of the nitrogen atoms-containing NH groups are hydrolyzed in the presence of moisture with the generation of surface $C=O$ groups and ammonia.

OXYGEN PLASMA TREATED ASPEN

Results from survey ESCA data indicate that the relative surface atomic composition of the extracted and oxygen plasma exposed aspen substrates exhibit comparable atomic composition to that of the extracted aspen substrates (C: 69.7% and O: 30.3%). However, the increased C1s and O1s Ar-CO-O peak surface areas of oxygen plasma treated samples (Figure 9a,b) relative to the extracted one, allow us to suggest that simultaneous surface functionalization and etching mechanisms developed during the oxygen plasma treatment. ATR-FTIR measurements also are indicative for the presence of aromatic C-H and unsaturated $C=C$ bonds in the plasma modified aspen structure (Figure 10a,b).

FIGURE 9. High resolution C1s and O1s ESCA diagrams of O$_2$-plasma treated aspen fibers (non-extracted).
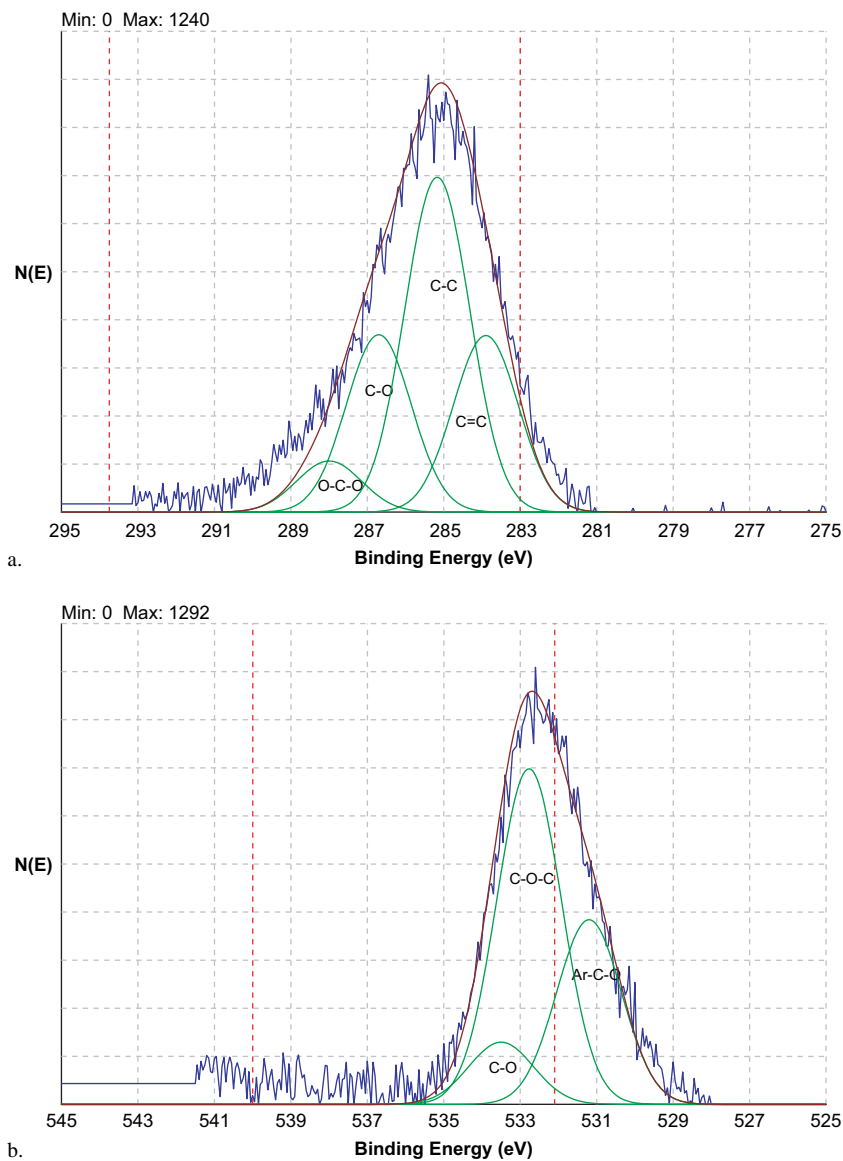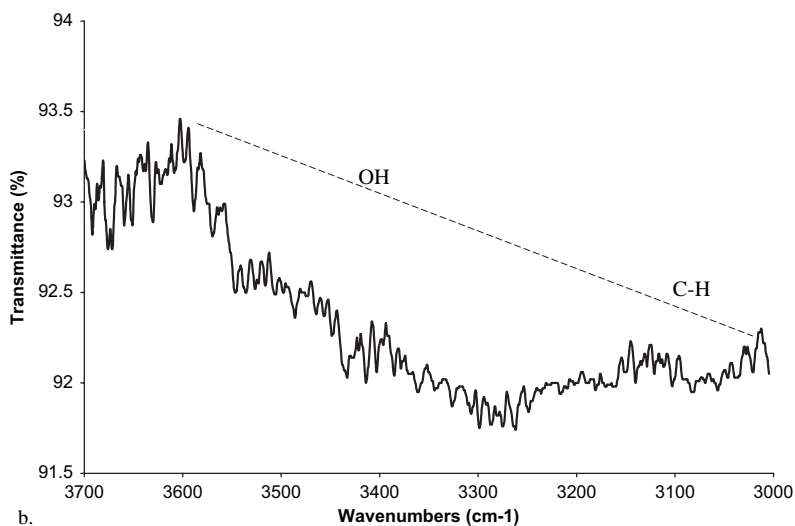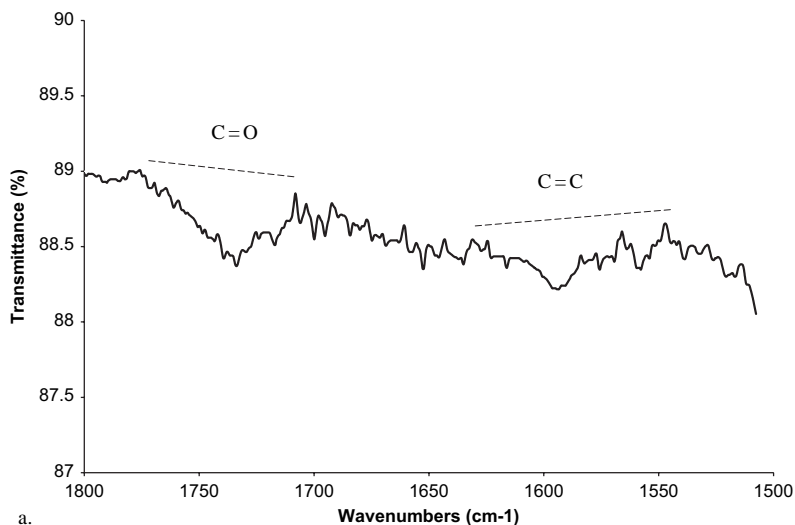
FIGURE 10. FTIR spectrum of O_2-plasma treated aspen fibers (extracted). Wavenumber ranges: (a) 1800–1500; (b) 3700–3000.

MECHANICAL TESTING

Specimens were tested for dry and wet strength according to ASTM Standard 1037–99. For wet strength, specimens were soaked in water at 20°C for 24 h and tested immediately upon removal from the water. Tests were run on four different specimens and the results averaged.

Figure 11a and b, show the load deflection curve for dry control and plasma treated fiberboards under dry and wet conditions. The highest dry strength was observed in the O_2 plasma treated non-extracted and extracted boards. Ammonia plasma-treated extracted fiberboards exhibit also a significantly increased strength. As expected, wet strength is the highest in the O_2- and ammonia-plasma treated and non-extracted fiber but the corresponding strength values are still significantly lower that the dry strength. It is suggested that limited water absorption (Table 2), due to the presence of hydrophobic extractives, renders diminished swelling of the fiberboards and as a result the achievement of lower strength values. It also should be noted that only the oxygen-plasma treatment resulted in positive swelling characteristics.

CONCLUSIONS

1. Ammonia- and oxygen-plasma treatments of virgin and extracted aspen fiber samples have been successfully accomplished.
2. Survey and high resolution ESCA results and ATR-FTIR data resulting from non-modified and modified Aspen fiber surfaces indicate that regardless of the nature of the plasma gases additional polar functionalities were surface-implanted onto the discharge-exposed substrates.
3. Fluorescamine test evaluations showed the absence of primary amine functionalities on the ammonia-plasma-treated fibers. It is suggested based on the analytical data that -N-H- and – NH-CO- groups are the main nitrogen-containing functionalities.
4. Removing the extractives from the surface of aspen fibers results in a 20% decrease in the relative surface atomic carbon concentration and in a proportional increase in oxygen content.
5. The influence of extractives on the cold-plasma treatment is significant.
6. Thickness swelling of the non-extracted and extracted aspen fiberboards was decreased in the oxygen-plasma treated samples, while little effect on the thickness swelling was observed in the ammonia-plasma exposed boards.
7. As Expected, wet strength of all fiberboards was lower than dry strength. O_2 plasma treatment of the non-extracted and extracted fiber

FIGURE 11. Load deflection curve for dry and wet control and plasma treated fiberboards: 1 = control Aspen fiberboard (non extracted & non plasma treated); 2 = extracted Aspen fiberboard (soxhlet extracted with Toluene: EtOH solution, non plasma treated); 3 = NH_3 – Plasma Aspen fiberboard (non extracted); 4 = NH_3 – Plasma Aspen fiberboard (soxhlet extracted with Toluene: EtOH solution); 5 = O_2 – Plasma Aspen fiberboard (soxhlet extracted with Toluene: EtOH solution); and 6 = O_2 – Plasma Aspen fiberboard (non-extracted).

a.
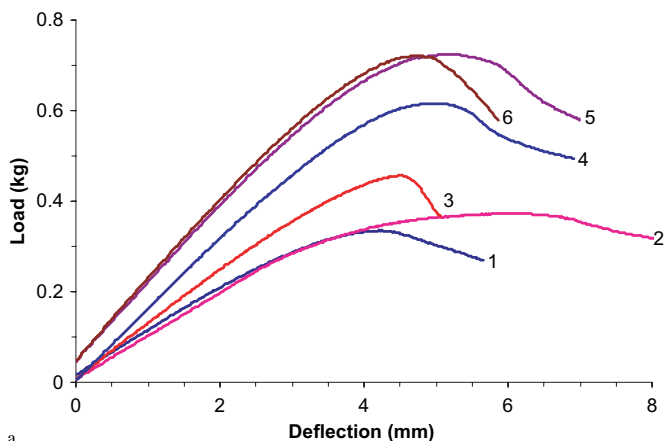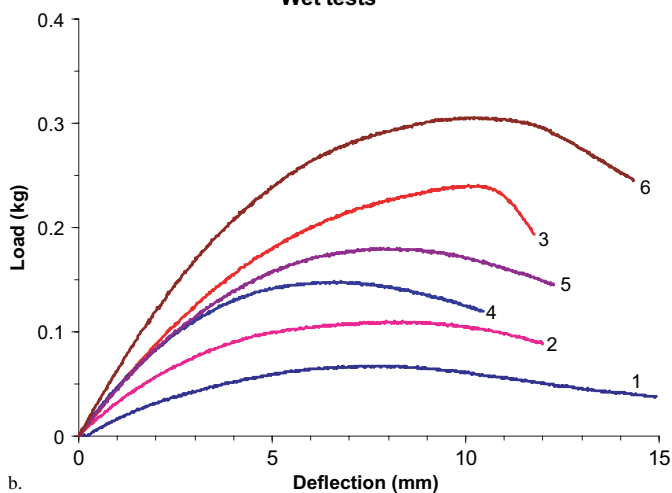
b.

resulted in the highest dry strength of the fiberboards, whereas O_2 plasma treatment of the non-extracted fiber resulted in the highest wet strength. Ammonia-plasma treatment of the extracted aspen boards also resulted in a significant increase of the strength values.

8. The influence of plasma-enhanced surface coating of aspen fibers with hydrophobic macromolecular layers on the water–swelling and mechanical properties of the resulting fiberboards will be evaluated in the future.

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